commercial resins show a high capacity but a poor selectivity towards metal ions.

The combination of the physical strength of an inorganic support with the higher ion exchange capacity and kinetics of the organic ion exchangers could, in the future, lead to an interesting class of ion exchangers with special applications.

See also: **II/Ion Exchange:** Historical Development; Inorganic Ion Exchangers; Organic Membranes; Theory of Ion Exchange.

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Organic Membranes

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Organic ion exchange membranes are made of insoluble polymeric foils, tubes or hollow fibres to which ion exchange groups are covalently bound. The membranes have all the properties typical of ion exchange resins and the ability to keep two different solutions physically separated. Thus, the main property of an ion exchange membrane is a selective exchange of ions and a selective permeability to ions, water or other specifically membrane-soluble species. Although optimizing all the properties would be difficult in the case of one membrane used for a variety of applications, the most desired property is always high membrane selectivity, which allows the separation of ions with low energy consumption and high transport rates. There are many additional requirements for ion exchange membranes, such as low electric resistance, high permselectivity, low free diffusion of salts (leakage), low osmotic water transport, high mechanical strength, high selectivity between ions of the same charge and high chemical stability.

Preparation

Ion exchange membranes can be classified as monopolar or bipolar. In parallel, some intermediate membranes have been categorized as mosaic and amphoteric. The monopolar membranes can be divided into cation exchange membranes and anion exchange membranes. Combination of these membranes results in a bipolar ion exchange membrane.

The properties of any ion exchange membrane are determined by the properties of its polymer matrix and the type and concentration of the fixed ionic moieties. The polymer matrix of ion exchange membranes is usually cross-linked. The degree of cross-linking extensively influences the degree of swelling (water sorption), chemical and mechanical stability and membrane permeability by changing ionic mobility inside the membrane phase. Thus, the proper selection of membrane components, their content and the method of preparation significantly affect the membrane properties and structure. The subsequent chemical modification of the polymer matrix involves the introduction of ionogenic groups, resulting typically in the following fixed sites:

Cation exchange membranes:

$$-SO_3^-, -COO^-, -PO_3^{2-}, -HPO_2^-, SeO_3^-$$

Anion exchange membranes:

$$-NH_3^+, -RNH_2^+, -R_3N^+, =R_2N^+, -R_3P^+, -R_2S^+$$

Monopolar Membranes

The simplest ion exchange membranes are composed of derivatives of styrene-divinylbenzene copolymers or vinylpyridine-divinylbenzene copolymers. In order to maintain their mechanical strength, these homogeneous membranes are often reinforced by backing materials. Other simple ion exchange membranes can be synthesized from finely powdered ion exchange resin and inert polymer powder applied as a bonding material. Nowadays, more advanced methods of membrane preparation are recommended. The most widely used methods are:

1. Impregnation of a basic polymer component with styrene and divinylbenzene for copolymerization followed by chemical modification (e.g. sulfonation);
2. Casting the mixed solution of poly(styrene sulfonic) acid and other inert polymer to produce an interpolymer or 'snake-in-cage' membrane;
3. Radiation grafting of a polymerizable monomer into a conventional polymer film and then introducing the ion exchange groups to the film. The films used are generally polyethylene, polypropylene and perfluoroethylene;
4. Chemical modification: this method involves direct introduction of ion exchange groups to a condensation-type polymer such as polysulfone, poly(ethylene oxide), poly(vinyl chloride), poly(vinylidene fluoride), poly(ether ether ketone). The membrane can be formed subsequently by casting the polymer solution, and then phase inversion. The procedure results in an anisotropic membrane structure with a thin skin layer and a supporting layer of sponge morphology;
5. Plasma polymerization. This relatively new method has been utilized to prepare: first, an anion exchange membrane by plasma polymerization of γ-aminopropylethoxydimethylsilane on a porous polymer film; second, a perfluorocarbon sulfonic acid membrane, by plasma polymerization of perfluorostyrene and SO_2; and third, a thin film sulfonic-type membrane, by plasma polymerization of ethylene and SO_2, or acetylene and SO_2.

Bipolar and Mosaic Membranes

Preparation methods of bipolar ion exchange membranes involve the introduction of cation exchange groups into one side of the membrane and anion exchange groups into the other side. In order to obtain the simplest bipolar membrane a separate cation exchange membrane and an anion exchange membrane can be glued together by using poly(styrene sulfonic) acid as a binder. Other bipolar ion exchange membranes have been prepared by graft polymerization of acrylic acid on one side of the porous polymer membrane and of *N*-(2-methacryoyloxyethyl)-*N*,*N*,*N*-trimethylammonium chloride on the other side, after oxygen plasma treatment of the porous membrane. The mosaic membranes are usually prepared by casting a multiblock copolymer composed of styrene, butadiene and vinylbenzyl dimethylamine and the subsequent introduction of cation and anion exchange groups into the polymer.

Structure

The properties of ion exchange membranes depend on their microstructure, which results from the differences in properties of their ionic and neutral components. The hydrocarbon ion exchange membranes are generally composed of derivatives of styrene-divinylbenzene copolymer and other inert polymers such as polyethylene and poly(vinyl chloride). Ionic derivatives of such copolymers are finely distributed in the inert polymer-forming microdomains because of their poor mutual compatibility. This inhomogeneity causes the distribution of ion exchange groups in the membrane material to be of varied local concentration. The local distribution of fixed charges can affect all the basic membrane properties, such as electrolyte sorption, electrical conductivity and permselectivity as well as mass transport rates. Specifically, a rather unusual structure is characteristic for perfluorocabon cation exchange membranes (with sulfonic or carboxylic acid groups), which can be classified as ionomer materials. Ionomers and ion-containing polymers with ionic sites and counterions spontaneously organized into dipole multiplets and, for some materials, into larger ion clusters containing 50 or more pairs of ions. These clusters are embedded in the perfluorocarbon membrane backbone, and are connected by narrow channels forming a continuous network.

Despite the heterogeneity resulting from the hydrophilic and hydrophobic nature of membrane components, certain additional morphology features originate from the method of preparation. So-called macroscopically homogeneous membranes can be prepared either by the polymerization of functionalized monomers or by careful modification of a homogeneous polymer film. However, depending on the method of preparation, the structural imperfection of membranes increases in the following order: interpolymer membranes, graft and block polymer membranes and membranes composed of powdered ion exchange resins embedded thereafter in an inert polymer binder.

General Physicochemical Properties

Ion exchange membranes are characterized by many of the parameters and properties described below. Independently, the basic properties describing equilibrium and transport properties of any membrane are as follows: ion exchange capacity (mol kg^{-1} of dried membrane), swelling (wt.% of water or other solvent), electrolyte sorption (mol kg^{-1} of water sorbed into membrane), distribution coefficient (ratio of solute concentration in the membrane and external solution), transference number for cations and anions (determined under standard conditions), electrical conductivity (or resistivity) and salt leakage (both determined after contacting the membrane with a standard electrolyte solution).

Co-ion Exclusion

In a cation exchange membrane, due to the system electroneutrality, the fixed anions are in equilibrium with mobile cations (referred to as counterions). In contrast, anions functioning as co-ions are more or less excluded from the membrane because their charge is identical to the fixed ion charge. This phenomenon, known as the Donnan exclusion of co-ions or electrolytes, enables the ideal membrane to transfer cations only. In the same way cations are excluded from the anion exchange membrane. The Donnan exclusion equilibrium, and thus the membrane selectivity, depends quantitatively on the concentration of the fixed ions, the valence of co- and counterions, the concentration of an equilibrating external solution, and the affinity of the exchange groups to respective counterions.

Transport Processes

The effectiveness of any membrane process is determined by the flux of species through the membrane. High fluxes arise because of high permeability of the internal aqueous membrane solution in respect to sorbed solutes. The presence and properties of transport-mediating functional ionic groups can either facilitate or hinder transport processes depending on many specific interactions between mobile species, the charged polyelectrolyte network and solvent. In general, membrane transport phenomena can be described by a general equation derived from the linear thermodynamics of irreversible processes:

$$J_{\mathrm{i}} = \sum_{i} L_{\mathrm{i,k}} X_{\mathrm{k}} \quad (\mathrm{i, k} = 1, 2, 3, \ldots, m)$$

where J_{i} denotes the flux of an individual permeant, the volume of solvent, heat or electricity transferred across a membrane, and $X_{\mathrm{i,k}}$ denotes the driving force represented by a difference in chemical potential ($\Delta\mu$), temperature (ΔT), pressure (Δp) or chemical affinity (A). $L_{\mathrm{i,k}}$ is the phenomenological coefficient linearly relating the flux and driving force. In practice, a general flux equation can be reduced to a description of a specific process by neglecting coupling transport phenomena. Depending on the solution and assumptions, various mathematical relations have been derived and applied to discuss transport rates of ionic substances and water through ion exchange membranes.

Permselectivity

The characteristic property of ion exchange membranes is their ionic permselectivity related to electrically driven processes and quantified by the following definitions:

$$P_{\mathrm{CEM}} = \frac{t_{+,\mathrm{CEM}} - t_{+}}{t_{-}} \text{ and } P_{\mathrm{AEM}} = \frac{t_{-,\mathrm{AEM}} - t_{-}}{t_{+}}$$

where t is the transference number for anions ($-$) and ($+$) cations in free electrolyte or acting as counterions (cation exchange membrane (CEM) and anion exchange membrane (AEM)) in an ion exchange membrane. An ideal permselective membrane should have a P value of 1, and the permselectivity is equal to zero when the transference numbers within the membrane are the same as in an electrolyte solution. Usually, transference numbers for practical membranes attain values from 0.8 to 0.98.

Separation Ability

One of the most important uses of ion exchange membranes is the selective separation of solutes. This can be performed because of the differences in the rates at which solutes permeate through the membrane. The size of this difference depends on the separation system, i.e. on the composition of feed solution and the membrane. The criterion of separation is that the composition of the mixture emerging from the membrane cell should be different from that entering on the feed side. Consequently, the separation factor α_B^A measures the extent of selective transport of A in relation to B:

$$\alpha_{\mathrm{B}}^{\mathrm{A}} = \frac{J_{\mathrm{A}} X_{\mathrm{B}}}{J_{\mathrm{B}} X_{\mathrm{A}}}$$

where J_{A} and J_{B} are the fluxes (transport rates expressed as numbers of moles issuing from the membrane per unit area and time) and X_{A} and X_{B} are the mole fraction of A and B in the feed.

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Membrane Processes and Applications

Many applications of ion exchange membranes in transport and separation processes have been made. Although the driving force for ions and water to penetrate through the membrane initially is primarily an electrochemical and chemical potential, it has been reported in recent years that the hydraulic permeability, temperature difference and difference in proton concentration can also be exploited. This results in some sophisticated transport mechanisms, the backgrounds and practice of which are reviewed below. The latest applications are due to developments in ion exchange membrane composition and structure. Ion exchange membranes with specific properties have been produced to meet many industrial requirements. For example, the following membranes of specific properties have been developed and commercialized: perfluorocarbon anion exchange membranes for high temperature usage and chemical stability in corrosive media, anion exchange membranes for diffusion dialysis (treatment of corrosive acidic waste waters), anion exchange membranes of high acid retention (electrodialytic concentration of dilute acids), hydrogen ion permselective cation exchange membranes (efficient electrodialysis) and monovalent cation or monovalent anion permselective membranes.

Diffusion Dialysis

Diffusion dialysis results from the difference in rates of permeation of salts and acids through anion exchange membranes. According to the scheme shown in **Figure 1**, this membrane allows the selective transport of anions across the membrane, ideally remaining impermeable to cations other than protons. Diffusion dialysis has been exploited to remove acids from solutions containing different cation species. High quality anion exchange membranes allow this method to be applied to the recovery of sulfuric and other acids from waste solutions generated in steel, metal-refining and electroplating industries.

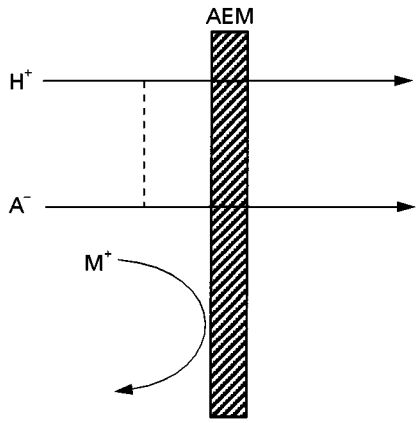

Figure 1 Scheme of diffusion dialysis of salt and acid through anion exchange membrane (AEM).

Donnan Dialysis

Donnan dialysis, also referred to as ion exchange dialysis, occurs after placing an ion exchange membrane between two solutions containing different electrolytes. According to the scheme depicted in **Figure 2**, ions can cross a membrane when their sign is opposite to the sign of the membrane-forming polyelectrolyte. At the same time, the permeation of co-ions is hindered because of their electrostatic exclusion from the membrane phase. Usually, divalent or univalent metal cations are transported from the dilute feed solution into the highly acidic stripping solution. The coupling of fluxes in this membrane system makes it possible to reach a stable flux of cations from dilute to concentrate phase. Thus, uphill transport (chemical pumping) arises as a result of the interdiffusion of different counterions.

The final distribution of ions between two membrane-adjacent solutions corresponds to the Donnan equilibrium principle, which in the typical case of M^{z+}/H^+ Donnan dialysis, takes the following form:

$$C_{M,s} = kC_{M,f}\left(\frac{C_{H,s}}{C_{H,f}}\right)^{Z_M}$$

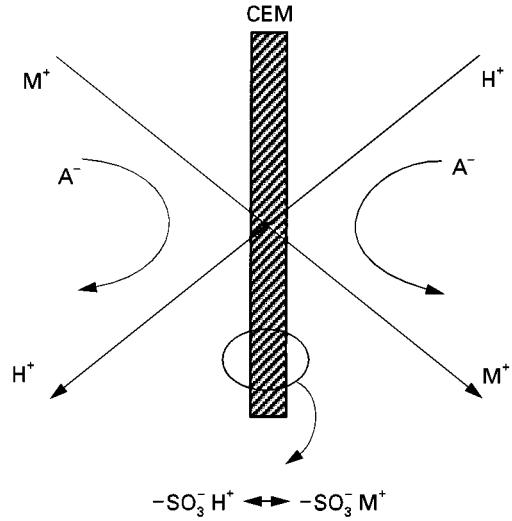

Figure 2 Scheme of Donnan dialysis of M^+ and H^+ cations through exchange membrane (CEM). (From Wódzki R, Szczepański P and Pawlowski M (1999) Recovery of metals from electroplating waste solutions and sludge. Comparison of Donnan dialysis and pertraction technique. *Polish Journal of Environmental Studies* 8(2): 111–124.)

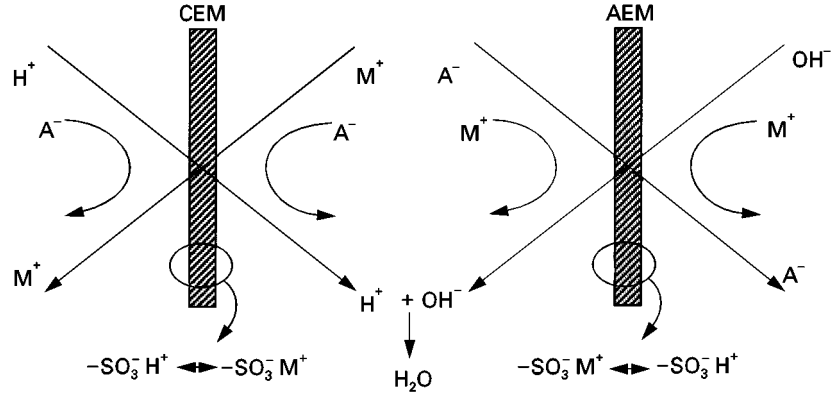

Figure 3 Scheme of neutralization dialysis of M^+ cations and A^- anions through cation (CEM) and anion exchange membranes (AEM).

Consequently, when the initial concentration (C) of metal ions (M) in the feed (f) is much lower than the concentration of counter-transported protons (H) in the stripping solution, it is possible to attain substantial enrichment of metal ions in the receiver, or alternatively, almost complete removal of these cations from the feed. Despite the many advantages, only a pilot dialyser for the nuclear industry (recovery of ^{134}Cs, ^{90}Sr and concentration of uranyl ions) has been reported as large scale implementation of this technique. On the other hand, Donnan dialysis is widely used in analytical laboratories as an efficient method for the preconcentration and separation of various cations and anions or for the treatment of complex matrices before analysis.

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Neutralization Dialysis

Neutralization dialysis is a membrane process based on the coupling of two simultaneously occurring Donnan dialyses. According to the scheme shown in **Figure 3**, in this case a salt solution is separated from an external acidic and basic solution with a cation and anion exchange membrane, respectively. Protons and hydroxide ions permeate into the desalination compartment by the Donnan dialysis mechanism, which generates the counter-flow of other cations and anions. Under ideal conditions, i.e. without salt leakage and with balanced fluxes of protons and hydroxide ions, the overall process results in almost complete desalination of the internal solution. The practical use of neutralization dialysis for the demineralization of mixtures containing some organic substances (mono-, oligo- and polysaccharides) and polyelectrolytes is recommended.

Pervaporation

In pervaporation processes the application of ion exchange membranes instead of inert polymer membranes is possible. During this process (**Figure 4**) an ion exchange membrane is kept in contact with a stream of the mixture of water and miscible organic solvent, whereas the second side of the membrane is kept under vacuum. Due to the strong affinity of ionic sites to water and an inertness of the polymer backbone to an organic component, water can permeate through the membrane. Thus, the permeation of water can be optimized by changing the membrane polymer, the type and content of the membrane-forming polyelectrolyte (ionomer, cross-linked copolymer, etc.), and the kind of counterions. For instance, cation and anion exchange membranes of various ionic forms have been examined for the separation of alcohol–water and pyridine–water mixtures.

Fixed-site Mediated or Carrier Transport

Analogous to liquid membrane transport mediated by the mobile carriers, ion exchange sites can be exploited as fixed carrier centres in the polymer membrane phase. The membranes exhibiting such a function are referred as fixed-site carrier membranes or reactive membranes. In general, transport phenomena in such membranes are believed to occur as a sequence of exchange reactions between permeating solute and reactive groups located along the polymer

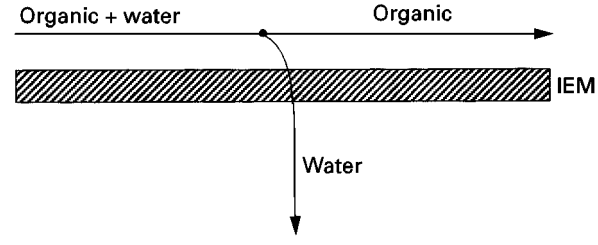

Figure 4 Scheme of water pervaporation through ion exchange membrane (IEM).

chain rather than by pure diffusion phenomena. To be effective the transport mechanism requires fixed sites to be mobile over a certain restricted area. Moreover, their concentration should exceed a certain concentration, enabling instantaneous overlapping of loaded and unloaded centres. Despite many difficulties in understanding and in the theoretical description of this type of membrane transport and separation, a number of processes have been developed. The most interesting are processes dealing with the separation of gaseous substances (CO_2), olefins and sugars. For example, a cation exchange membrane exchanged with ethylenediamine is selectively permeable to CO_2 (from a feed containing CH_4 and H_2S). On the other hand, silver ions exchanged with a cation exchange membrane form an Ag^+–olefin complex in the membrane phase. Carrier transport of 1-hexene and 1,5-hexadiene from a decane phase through a cation exchange membrane impregnated with silver ions has been reported. Similarly, styrene permeates selectively (compared with ethylbenzene) through the Ag^+ form of cation exchange membrane. A selective transport of sugars via complexation with borate ions fixed in an anion exchange membrane has been performed and referred as carrier–relay transport. In this way, the selective transport of D-glucose, D-xylose, D-arabinose, D-mannose, D-galactose, D-fructose, L-sorbose, sucrose and D-lactose can be achieved.

Membrane Extraction–Hybrid Membrane Systems

The combinations of liquid membrane extraction (usually mediated by specific ion exchange extractant/carrier) with ion exchange membranes in some relatively new membrane systems have been called membrane hybrid systems (MHS). The scheme of cation transport in a simple MHS is depicted in **Figure 5**. The MHS operation involves a series of ion exchange–diffusion processes (ion exchange membrane) as well as permeation through a liquid membrane. The presence of an ion exchange membrane at a particular interface stabilizes the liquid membrane and enhances the interfacial ion exchange reactions because of high, and specific, sorption of certain ions by the membrane polyelectrolyte. The long term and stable operation of a liquid membrane system can be achieved with the MHS idea. The selective and active transport of metal cations, inorganic anions and some carboxylic acids has been reported. It is worth noting that these systems can be regarded as biomimetic, which means that they correspond to a cellular envelope of Gram-positive bacteria composed of an ion exchange polymer membrane (cell wall) and a quasi-liquid membrane (cytoplasma membrane).

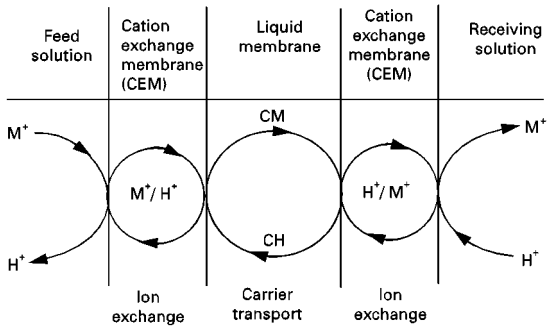

Figure 5 Scheme of membrane hybrid system composed of two cation exchange membranes and the liquid membrane containing an ionic carrier (C). Coupled countertransport of M^+ and H^+ cations. (From Wódzki R, Szczepański P and Pawlowski M (1999) Recovery of metals from electroplating waste solutions and sludge. Comparison of Donnan dialysis and pertraction technique. *Polish Journal of Environmental Studies* 8(2): 111–124.)

Membrane Permeation Coupled to External Reaction

It is possible to accelerate chemical reactions by removing some products from the reaction medium before the reaction reaches its equilibrium state. Ion exchange membranes are effective in these cases. For example, fermentation processes producing ionic materials such as acetic acid can be carried out continuously as an extractive fermentation by removing the products by means of diffusion dialysis, pervaporation or electrodialysis. Ion exchange membranes can be applied to improve the kinetics of chemical reactions by the pervaporative removal of water from the reaction medium. Such a technique has been demonstrated for esterification of oleic acid with ethanol and of propionic acid with isopropanol or propanol.

In more advanced systems, membrane transport phenomena are coupled with enzyme reactions. The most representative system is composed of a cation exchange membrane layer, a porous membrane layer containing entrapped urease and an anion exchange membrane layer. Urea is decomposed into NH_4^+ and CO_3^{2-} in the enzyme layer and products permeate through the respective membrane layers without application of an electric field. A similar idea is utilized in many enzymatic membrane sensors.

Ion-Exchange Membrane Separators in Power Sources, Sensors and Electrodes

A typical example is the use of an ion exchange membrane as a solid polyelectrolyte for a fuel

cell. The composite, in which anode catalyst, poly(fluorocarbon sulfonate) cation exchange membrane and cathode catalyst are combined, has been used for hydrogen-oxygen fuel cells. The only product of this cell is water, which seems to be desirable from an ecological point of view. Methanol has also been reported for the generation of electricity and utilizable industrial chemicals by the use of a membrane-containing fuel cell. Various applications of ion exchange membranes of low electrical resistance in alkali batteries are possible. Modern cation exchange membranes made of acrylic acid grafted onto polyethylene film are widely used as separators in alkaline batteries, such as a Ni–Cd secondary battery.

Water content of the ion exchange membranes and thus their physicochemical properties changes with humidity. Therefore, ion exchange membranes are usable as a working part of a hygrometer. When water content of the membrane increases with increasing humidity, the increase in the current or ionic conductivity between the electrodes on both sides of the membrane can be measured and calibrated.

The concentration of alcohol can be indirectly determined by means of a membrane with fixed alcohol-dehydrogenase or alcohol-oxydase. It has been reported that these sensors operate accurately in acidic media after coating the enzyme-fixed membrane by a cation exchange membrane (organic acids cannot approach the enzyme membrane). A similar concept is used for constructing *in vivo* operating sensors for glucose presence and concentration. Some modified electrodes are constructed by coating classic electrodes with layered ion exchange materials. A typical example is the perfluorocarbon cation exchange membrane-coated electrode to control the permeability of redox species such as $Ru(bpy)_3Cl_3$ or 1,1-dihydroxymethylferrocene.

Industrial Applications

Membrane Electrodialysis

The basic principle of membrane electrodialysis is presented in **Figure 6**. The membranes are arranged in a series of anion and cation exchange membranes placed between an anode and a cathode. The cations migrating towards the cathode permeate the negatively charged cation exchange membrane and are retained by anion exchange membranes. The anions are transported in the opposite direction. The final result is an increase in ion concentration and ion depletion in alternate compartments. Membrane electrodialysis has been developed within the last several years, mainly for desalting brackish waters and concentrating brine from seawater. Nowadays,

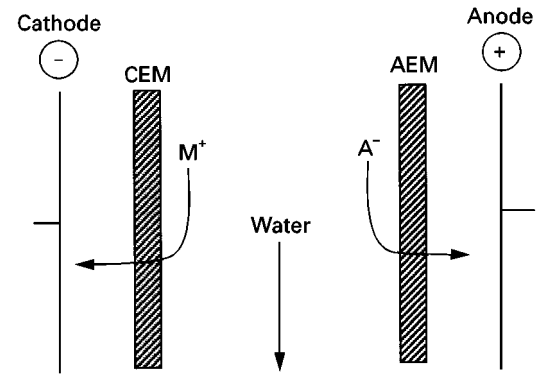

Figure 6 Basic idea of membrane electrodialysis.

electrodialysis is widely applied in environmental protection (depolluting and recycling of chemicals), in bio-industries (food, pharmacy and biotechnology) and in the treatment of drinking water. Some of these new applications have led to substantial improvements in membrane quality. For instance, special membranes with low permeability to the divalent ions in respect to the monovalent ones, membranes with very low permeability to hydroxyl ions or very low permeability to protons, are currently produced.

Membrane Electrolysis

A typical example of this kind of application is the membrane electrolysis of sodium chloride to produce chlorine, hydrogen and sodium hydroxide (membrane chlor-alkali process). A schematic diagram for this process is presented in **Figure 7**. Perfluorocarbon cation exchange membranes for chlor-alkali processes should have an anisotropic structure in their cross-section. The cathode side of the membrane has a thin layer of carboxylic acid groups of a given ion exchange capacity, and the anode side of the membrane has a thick sulfonic acid group layer or a carboxylic acid group layer of high ion exchange capacity. Ion exchange membranes can also be applied for the electrodialysis of water to produce hydrogen and oxygen. The technology exploiting a solid polymer electrolyte method is efficient when perfluorocarbon cation exchange membranes are used in the form of a stack with catalytic electrodes covering the membrane surfaces. Ion exchange membranes are also used as separators in organic synthesis by electrolysis. A typical example is the hydrodimerization of acrylonitrile to produce adiponitrile.

Conclusion

The technique of membrane separation can be considered as an energy-saving method because, in

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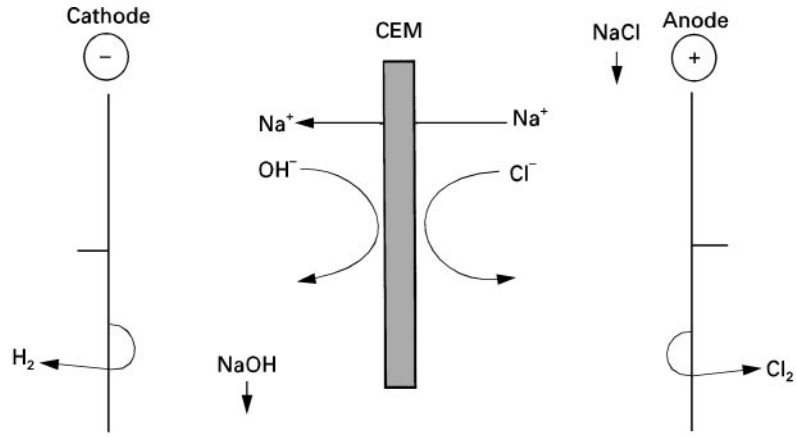

Figure 7 Chlor-alkali process (membrane electrolysis) using a cation exchange membrane (CEM).

general, it does not cause phase conversion. From this point of view, ion exchange membranes and their application technology are one of the most advanced methods enabling the embodiment of closed-loop chemical processes. As solid polyelectrolytes, the membranes are easy to regenerate, recycle and/or promote continuous usage to improve industrial processes. Environmental benefits can be achieved as well.

Since the use of ion exchange membranes has become very diverse, the requirements for membranes with new properties will increase. These requirements should result in the development of new highly functionalized ion exchange membranes. Photosynthetic membranes, biomimetic membranes, complex and specialized membrane sensors, polymerized phospholipids and polymerized crown ethers with ionic groups can be considered as new possible areas of membrane science development.

See also: **II/Ion Exchange:** Catalysis: Organic Ion Exchangers; Historical Development; Inorganic Ion Exchangers; Organic Ion Exchangers; Theory of Ion Exchange. **III/Porous Polymer Complexes for Gas Separations: Membrane Separations**.

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Phosphates: Novel Layered Materials

See **II / ION EXCHANGE / Novel Layered Materials: Phosphates**